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IS 33 (1992): Inorganic Pigments and Extenders for Paints –
Methods of Sampling and Test [CHD 20: Paints, Varnishes and
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भारतीय मानक

रंग रोगन के लिए अकार्बनिक वर्णकों और विस्तारकों के
नमूने लेने और परीक्षण की पद्धतियाँ
(तीसरा पुनरीक्षण)

Indian Standard

INORGANIC PIGMENTS AND EXTENDERS
FOR PAINTS — METHODS OF SAMPLING AND
TEST

(Third Revision)

First Reprint JULY 1997

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

AMENDMENT NO. 1 JUNE 2003
TO
**IS 33 : 1992 INORGANIC PIGMENTS AND EXTENDERS
FOR PAINTS — METHODS OF SAMPLING AND TEST**

(*Third Revision*)

(*Page 2, clause 5.3.2*) — Substitute the following for the existing clause:

'5.3.2 For ascertaining the conformity of the lot to the requirement of any specification, tests shall be carried out for each lot separately. The number of packages to be selected shall be 10 percent of total packs if number of packs are 100 or less than 100 and shall be 5 percent of total packs in case of number of packs are more than 100.'

(*Page 3, clause 9.1.1*) — Substitute the following for the existing:

9.1.1 Sieve — IS sieve of desired micron size [*see IS 460 (Part 2)*].'

(*Page 5, clause 11.0, line 1*) — Substitute 'coloured pigments/extenders' for 'coloured pigment'.

(*Page 6, clause 11.3.2.6*) — Insert the following new clause after 11.3.2.6:

'11.4 Test for Extenders

Mix the extender with binder to produce paste consistency using glass or marble plate. Simultaneously, also prepare the paste of approved sample of same apparent consistency.

Compare the colour of the sample with that of the approved sample of extender as described in 11.3.1.6.'

(*Page 12, clause 17.2, line 2*) — Substitute 'Dry-sieve' for 'Dry, sieve'.

(CHD 20)

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FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1950 and was primarily based on the interim coordinated draft complied with the assistance of all concerned by the Coordinating Subcommittee of the No. 5 Standing Committee on Specifications for Paints and Allied Stores of the General Headquarters (now Army Headquarters), India. It was subsequently revised in 1963 and 1976. In these revisions the test methods were updated and brought at par with the prevailing technology.

In this third revision, the methods have been further revised to bring them in line with the various parts of ISO 787 'General methods of test for pigments and extenders' issued by the International Organization for Standardization. Methods of test prescribed for the various requirements are technically equivalent to the corresponding methods given in ISO 787. However, methods for ether extract, ash content, lead restriction and freedom from lead continue to be the same without any change.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'.

Indian Standard

INORGANIC PIGMENTS AND EXTENDERS FOR PAINTS — METHODS OF SAMPLING AND TEST

(Third Revision)

1 SCOPE

This standard prescribes methods of sampling and test applicable to several inorganic pigments and extenders in dry condition.

2 REFERENCES

The Indian Standards listed in Annex A are the necessary adjuncts to this standard.

3 TERMINOLOGY

3.0 For the purpose of this standard, the definitions given in IS 1303 : 1983 and the following shall apply.

3.1 Freedom from Lead (Lead-Free)

It means that the material does not contain lead or compounds of lead or a mixture of both, calculated as metallic lead (Pb), exceeding 0.03 percent by mass.

3.2 Standard Atmospheric Conditions for Testing

An atmosphere with a relative humidity of 65 ± 5 percent and a temperature of $27 \pm 2^{\circ}\text{C}$ provided that in a given series of experiments, the temperature does not vary by more than $\pm 1^{\circ}\text{C}$ (see also IS 196 : 1966).

3.3 Room Temperature

A temperature between 21 and 38°C , unless otherwise specified.

4 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1992) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

5 SAMPLING

5.1 General

In drawing samples, the following precautions and directions shall be observed:

- a) Samples shall not be taken in an exposed place;

- b) The sampling apparatus shall be clean and dry;
- c) Samples, the material being sampled, the sampling apparatus and the containers for samples shall be protected from adventitious contamination;
- d) A representative sample shall be drawn after the contents of each container selected for sampling are mixed thoroughly;
- e) The samples shall be placed in clean, dry and air-tight metal or opaque glass containers on which the material has no action;
- f) The sample containers shall be of such a size that they are almost completely filled by the sample;
- g) The sample containers shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the month and year of manufacture of the material; and
- h) Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

5.2 Sampling Apparatus

Sampling shall be carried out by means of a suitable sampling scoop of the type shown in Fig. 1. The scoop shall be made of metal and shall have a semicircular (or C-shaped, if found suitable) cross-section.

5.3 Scale of Sampling

5.3.1 Lot

In any consignment of one type of pigment or extender, all the packages of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment of one type of pigment or extender is known to consist of different batches of manufacture or of different size of packages, then the packages of the same size and belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

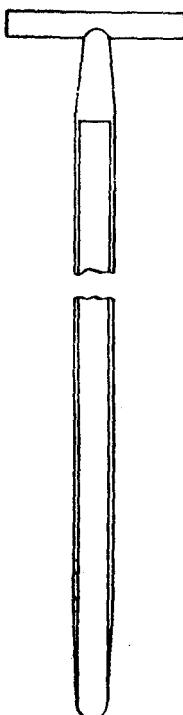


FIG. 1 SAMPLING SCOOP

5.3.2 For ascertaining the conformity of the lot to the requirement of any specification, tests shall be carried out for each lot separately. The number of packages to be selected at random from various sizes of lots shall be in accordance with Table 1.

Table 1 Scale of Sampling

Lot Size (1)	Sample Size (2)
Up to 50	2
51 ,, 100	3
101 ,, 300	4
301 ,, 500	5
501 ,, 1 000	7
1 001 ,, 3 000	10
3 001 and above	15

5.3.3 The packages shall be selected at random and to ensure randomness of selection, the following procedure is recommended for use:

'Starting from any package in the lot, count them in any suitable order as 1, 2, 3..... up to r and so on, where r is the integral part of

N/n (N being the lot size, and n the corresponding sample size given in Table 1). Every r th package thus counted shall be withdrawn to give sample for tests'.

5.4 Preparation of Test Samples

5.4.1 From each of the packages selected according to 5.3.3, a small representative portion of the material shall be drawn with the help of the sampling scoop by inserting it into the material at different depths. (The approximate quantity of material to be drawn from a package shall be not less than 500 g).

5.4.2 Out of these portions, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determinations for all the characteristics specified under 5.5. If the quantities of material mixed from different portions is much larger, the process of coning and quartering may be adopted to obtain the requisite quantity (see Note). The composite sample shall be divided into three parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

NOTE—On a clean impervious surface, heap the sample into a cone by depositing separate small quantities, one on top of the other, ensuring that the apex always remains in the same vertical line. Break down the cone, mix and reform the cone twice in the same manner. Flatten the third cone by pressing with a metal or glass sheet so that a mass of uniform thickness and diameter is obtained and quarter along two diameters. Discard the diagonally opposite quarters and heap remaining two quarters into a cone. Repeat this process until a sufficient quantity is left to provide sample of not less than 500 g.

5.4.3 The three parts of the composite sample shall be transferred to separate containers. These containers shall be sealed airtight and labelled with full identification particulars given in 5.1(g).

5.4.4 The referee test sample shall bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier, and should be used in case of any dispute between the two.

5.5 Number of Tests

Tests for determination of all the characteristics specified in the relevant material specification shall be conducted on the composite sample.

5.6 Criteria for Conformity

A lot shall be declared as conforming to the requirements of the specification if the characteristics tested on the composite sample satisfy the requirements given in the material specification.

6 FORM AND CONDITION

Unless specified otherwise, the material shall be supplied in the form of dry powder or in such a condition that it can be reduced to the powder form by crushing, without grinding action, under a palette knife.

7 PRELIMINARY EXAMINATION OF SAMPLE

The material shall be visually examined for the presence of foreign matter and visible impurities.

8 VOLATILE MATTER

8.0 General

The expressed loss in weight on heating the material to constant mass at $105 \pm 2^\circ\text{C}$ as percent will give the volatile matter content of the sample.

8.1 Apparatus

8.1.1 Weighing Bottle

Squat form, wide-mouthed, with ground glass stopper, about 30 mm in height and 70 mm in diameter.

8.1.2 Oven — Capable of being maintained at $105 \pm 2^\circ\text{C}$.

8.1.3 Balance — Accurate to 1 mg or better.

8.1.4 Desiccator — Containing an efficient desiccant.

8.2 Procedure

Spread 5 ± 0.5 g of the sample having volatile matter more than 1 percent and high bulk density (10 ± 1 g for other samples) in a uniform layer on the bottom of the weighing bottle. Insert the stopper and weigh to the nearest 1 mg.

Heat the weighing bottle and contents, with the stopper removed, in the oven at $105 \pm 2^\circ\text{C}$ for a minimum of one hour. Allow to cool in the desiccator, insert the stopper and weigh to the nearest 1 mg. Repeat the heating for at least 30 minutes, allow to cool in the desiccator, insert the stopper and again weigh to the nearest 1 mg. Repeat this procedure of heating and cooling until the two successive weighings do not differ by more than 5 mg. Record the lower mass.

If the results of the two determinations differ by more than 10 percent of the higher value, repeat the whole procedure.

NOTE — If the material under test is unstable at 105°C , the test conditions should be agreed to between the purchaser and the supplier.

8.3 Calculation

Volatile matter, percent by mass

$$= \frac{100 (M_1 - M_2)}{M_1}$$

where

M_1 = mass, in g, of the sample taken for test; and

M_2 = mass, in g, of the residue.

9 RESIDUE ON SIEVE

9.0 General

The residue on sieve from a sample of pigment or extender will be determined by dispersing the material in water and sieving through the required micron sieve.

NOTE — This method is not suitable for determination of residue on sieve for carbon black and graphite.

9.1 Apparatus

9.1.1 Sieve — IS sieve of 45 micron [see IS 460 (Part 2) : 1985].

9.1.2 Brush — hog bristle, approximate dimensions 5 mm thick, 20 mm wide, 35 mm long.

9.1.3 Sintered Glass Crucible — of porosity Grade P 40 (see IS 12305 : 1988).

9.1.4 Oven — capable of being maintained at $105 \pm 2^\circ\text{C}$.

9.1.5 Balance — accurate to 1 mg or better.

9.1.6 Desiccator — containing an efficient desiccant.

9.1.7 Wash Bottle — containing the solution used for dispersing the test portion.

9.2 Procedure

9.2.1 Weigh, to the nearest 0.1 g, into a beaker of suitable capacity, a quantity of the sample which will give a sufficient amount of residue on the sieve. Generally, a test portion of 10 to 100 g is necessary, but in the case of products yielding a very low residue on the sieve a larger test portion, up to 1 000 g may be used.

9.2.2 Disperse the sample in a suitable volume of water (about 300 to 600 ml) containing, if required, a suitable dispersing agent (see Note 1). If the product specification recommends that mechanical assistance is generally required to achieve thorough dispersion, a stirrer should be used. It is recommended that the rotation of the stirrer shall not exceed 500 ± 50 revolutions per minute. The use of a mechanical stirrer shall be stated in the test report.

NOTES

1 The quantity of the dispersing agent should be between 0.2 and 0.5 percent of the mass of the sample. The type and quantity of the dispersing agent to be used should be agreed to between the supplier and the purchaser.

2 It is important that the dispersion of the pigment or extender in the aqueous medium should be thorough and that no flocculation should occur during the determination.

9.2.3 Pour the dispersion, if necessary, in portions through the sieve. With the aid of the wash bottle filled with the solution used to disperse the sample, rinse the beaker and pour all the rinsings through the sieve. Wash the sample with the same solution until the washings passing through the sieve are clear and free of dispersed material. During this process, the duration of which should not exceed 5 min, care should be taken to avoid the destruction of agglomerates. Finally, wash any adherent particles with the brush into the sieve and wash the residue on the sieve with water to free it from the dispersing agent.

9.2.4 Treat the residue on the sieve following one of the procedures given in **9.2.4.1** to **9.2.4.4**.

9.2.4.1 Wash the residue into the previously heated and weighed sintered glass crucible and dry in the oven at $105 \pm 2^\circ\text{C}$ for one hour. Allow to cool in the desiccator and weigh to the nearest 1 mg. Repeat the heating for at least 30 min, allow to cool in the desiccator, insert the stopper and again weigh to the nearest 1 mg. Repeat this procedure until two successive weighings do not differ by more than 5 mg. Record the lower mass.

9.2.4.2 Transfer the residue with distilled water into a previously heated and weighed 50-ml beaker. Evaporate the water and dry in the oven at $105 \pm 2^\circ\text{C}$ for one hour. Continue as described under **9.2.4.1** above.

9.2.4.3 Dry the residue on the sieve in the oven at $105 \pm 2^\circ\text{C}$ for one hour. Transfer the residue into the previously heated and weighed weighing bottle and weigh to the nearest 1 mg. Repeat the heating for at least 30 min, allow to cool in the desiccator, insert the stopper and again weigh to the nearest 1 mg. Repeat this procedure until two successive weighings do not differ by more than 5 mg. Record the lower mass.

9.2.4.4 In case of very low residue on the sieve, use small sieve of diameter approximate 80 mm and follow the procedure given under **9.2.3**, using a sieve previously dried to constant mass at $105 \pm 2^\circ\text{C}$. Dry the residue on the sieve in the oven at $105 \pm 2^\circ\text{C}$ for one hour. Continue as described under **9.2.4.1**.

9.3 Calculation

$$\text{Residue on sieve, percent by mass} = \frac{100 \times m}{M}$$

where

m = mass, in g, of residue; and

M = mass, in g, of the sample.

10 OIL ABSORPTION**10.0 General**

The quality of refined linseed oil absorbed by a sample of pigment or extender is determined and expressed on mass/mass basis.

10.1 Apparatus**10.1.1 Plate**

Ground glass or marble, at least 300 mm \times 400 mm.

10.1.2 Palette Knife

Tapered steel blade of the approximate dimensions 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

10.1.3 Burette

Capacity 10 ml (see IS 1997 : 1982).

10.1.4 Balance — With an appropriate accuracy.**10.2 Reagent**

Refined linseed oil, having an acid value of 7.5 to 8.5 (see IS 75 : 1973).

10.3 Sample

The quantity of the sample to be taken for test depends upon the expected oil absorption value. Table 2 gives the values regarding the amounts of sample required for different ranges of the expected oil absorption value.

Table 2 Quantity of Sample to be Taken for Oil Absorption Test

Expected Oil Absorption Value ml/100 g (1)	Mass of the Sample g (2)
Less than 10	20
10 to 30	10
31 to 50	5
51 to 80	2
Over 80	1

10.4 Procedure

Place the sample on the plate. Add the linseed oil slowly, 4 or 5 drops at a time, from the burette. After each addition, rub the oil into the product with the palette knife and continue the addition of oil at this rate until conglomerates of oil and product are formed. From this point, add the oil 1 drop at a time and follow each addition of oil by thoroughly rubbing with the

palette knife. Cease the addition of oil when a paste of smooth consistency has been formed. This paste should just spread without cracking or crumbing and should only just adhere to the plate.

Read the burette and note the quantity of oil used. The time taken for the complete operation should be between 20 and 25 min which shall be utilized by the operation to manipulate the whole product mass with maximum effort.

Where a comparison is required with the oil absorption value of an agreed sample of product, repeat the test in exactly the same way using the agreed sample.

10.5 Calculation

The oil absorption value, expressed in mass per 100 g of product = $\frac{93V}{m}$

where

V = volume, in ml, of oil required; and

m = mass, in g, of the test portion.

NOTE — When the oil absorption value is to be expressed in ml of oil in 100 g of the product then use the following formula:

$$\frac{100V}{m}$$

11 COLOUR

11.0 General

The colour of a coloured pigment is compared with that of an approved sample.

11.1 Apparatus

11.1.1 Palette Knife

Tapered steel blade of approximately 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point, or a palette knife of a suitable plastic material.

11.1.2 Substrate — of minimum area 150 mm \times 50 mm.

Choose a substrate according to the binder used and the method of colour comparison. If a glass panel is used, it shall be clear and colourless.

11.1.3 Burette

With a delivery such that 1 ml of the binder contains about 35 drops.

11.1.4 Muller

Either of the following may be used.

11.1.4.1 Automatic muller

With ground glass plates, preferably water cooled, of diameter 180 to 250 mm, to which a variable but known force of up to about 1 kN

may be applied, the driven glass plate shall have a rotational frequency of between 70 and 120 rpm and the apparatus should have an arrangement for pre-setting the number of revolutions in multiples of 25.

NOTE — If the automatic muller does not have water cooled plates, care should be taken that temperature variations do not occur during the grinding operation.

11.1.4.2 Hand muller — With a diameter of 70 to 75 mm.

11.1.5 Plate, Ground Glass or Marble

For use when the automatic muller is not available.

11.2 Binder

The binder used shall be agreed between the purchaser and the supplier. If no binder is specified or agreed, linseed oil, complying with the requirements of the refined grade specified in IS 75 : 1973 shall be used.

11.3 Procedure

11.3.1 Using an Automatic Muller

11.3.1.1 Take a quantity of pigment for the test such that, when mixed with a sufficient quantity of the binder to obtain the dispersion, the resulting paste extends almost to the edges of the plates of the muller. Weigh the sample to the nearest 1 mg.

11.3.1.2 Transfer the sample to the clean lower plate of the automatic muller. Run a number of drops of the binder from the burette on to the blade of the palette knife and, using the blade, mix the binder and pigment. Add more drops of binder as necessary, to produce a paste of suitable consistency for milling.

11.3.1.3 Spread the paste in a band approximately 50 mm wide, about half-way between the centre and rim of the lower plate and clean the palette knife by drawing it across the upper plate. Close the muller plates, apply a force of about 1 kN and grind the paste in stages of 50 revolutions for each stage, picking up the paste with the palette knife and returning it to the 50 mm wide band after each stage.

NOTE — The applied force and the number of stages depend on the pigment tested, and should be the same for the pigment under test and the agreed sample.

11.3.1.4 When the grinding has been completed, add few more drops of the binder to obtain a suitable consistency, close the muller plates and grind the paste for a further 25 revolutions. Remove the paste from the plate and store it.

11.3.1.5 Take a similar amount of the approved sample of pigment and prepare a paste in the same way at a consistency equivalent to that

used in treating the sample, even if there is a variation in the quantity of binder required to achieve this consistency.

11.3.1.6 Colour comparison

Compare the colour of the sample with that of the approved sample by spreading the two prepared pastes in the same direction on the substrate in opaque strips not less than 25 mm wide with touching edges not less than 40 mm long. Compare the colour by examining the strips in diffuse daylight on the surface through the glass, immediately after application. Where good daylight is not available make the comparison in artificial daylight.

NOTE — By agreement between the supplier and the purchaser, a suitable colorimeter may also be used for making the comparison.

11.3.2 Using a Head Muller or Palette Knife

11.3.2.1 Weigh accurately between 0.1 and 1.0 g of the sample depending on the oil absorption value of the pigment under test.

11.3.2.2 Transfer the sample to the glass or marble plate. Run a number of drops of the binder from the burette on to the blade of the palette knife and using the blade, mix the binder and pigment. Add more drops of binder as necessary to produce a paste with a suitable consistency for milling.

11.3.2.3 When the pigment has become uniformly wetted with binder, start rubbing with the palette knife or hand muller using a backward and forward motion. The rubbing should spread the mixture over an area approximately 200 mm \times 575 mm. After 100 rubbing scrap the pigment binder mixture into a heap at the centre of the plate, making sure that any unground pigment is removed from the knife blade.

11.3.2.4 Repeat the rubbing operation using a further 100 rubbings and then add few more drops of the binder to obtain a suitable consistency. Mix well until the paste is homogeneous, and transfer it to one corner of the plate. Clean the rest of the plate.

11.3.2.5 Take similar amount of the approved sample of pigment and prepare a paste in the same way at a consistency equivalent to that used in treating the sample, even if there is a variation in the quantity of the binder required to achieve this consistency.

11.3.2.6 Colour comparison

Compare the colour of the sample with that of the approved sample of pigment as described in 11.3.1.6.

12 FASTNESS TO LIGHT

12.0 Two methods are given for determination of light fastness, one in artificial light source and the other in natural light source.

12.1 Artificial Light Source

12.1.1 Apparatus

12.1.1.1 Fadeometer

It shall have the following essential features:

- a) *Source of light* — A carbon arc composed of carbon electrodes enclosed in a heat-resisting glass globe to produce a complete spectrum from 279 nm through 1 200 nm. The arc shall operate on a direct current of approximately 11 to 13 amperes or an alternating current of 50 to 60 c/s and approximately 13 to 17 amperes, with 110 to 140 volts across the arc;
- b) The test specimens are vertically hung in test rack on the inner wall of a cylinder (rotating or fixed type), concentric with the arc. The distance of the test specimens shall be approximately 250 mm from the arc. No part of the specimen shall be more than 100 mm above or below the horizontal plane passing through the centre of the arc when it is fitted with new electrodes.
- c) The air inside the chamber shall be humidified by placing water in a trough or water-soaked cotton wicks in the chamber. A fan or blower unit maintains a constant flow of fresh air over the specimens; and
- d) A black panel thermometer placed in a rack in the chamber shall be used to monitor the temperature which shall not exceed 74°C.

12.1.2 Procedure

12.1.2.1 Preparation of panel

Mix the paste as prepared in 11.3.1.2 and 11.3.2.2 in a glass or marble pestle and mortar with sufficient quantity of boiled linseed oil (see IS 77 : 1976) or use the medium as agreed to between the purchaser and the supplier and not more than 5 percent of petroleum hydrocarbon solvent (Grade 145/205 of IS 1745 : 1978) or oil of turpentine and with paint driers (see IS 385 : 1979) if necessary, to obtain a consistency suitable for application by brushing. This operation shall take not more than 10 minutes. Brush the paint so prepared on a tinned steel panel and allow to hard dry.

12.1.2.2 Allow the panel prepared (see 12.1.2.1) to dry for 48 h and then expose for 72 h to the action of ultraviolet light in the fadeometer.

12.1.2.3 The sample shall be considered to have passed the test if there is no undue change of colour.

NOTE — In this test, xenon arc fadeometer may also be used.

12.2 Natural Light Source

12.2.1 Apparatus

12.2.1.1 Substrate

Cardboard panels of suitable size for the applicator used, of rigid quality and with a white high gloss lightfast, coated nonabsorbent surface suitable for the application of paint.

12.2.1.2 Film applicator or other device

Suitable for applying two films side by side of wet thickness 50 to 100 μm .

12.2.1.3 Cover sheet

Aluminium foil or other suitable opaque material.

12.2.1.4 Geometric grey scale — Complying with IS 768 : 1982.

12.2.1.5 Source of light

Natural daylight but artificial sources of light may also be used by agreement between the purchaser and the supplier.

NOTE — A mercury vapour lamp is not suitable for this test.

12.2.2 Procedure

12.2.2.1 Preparation of test specimen

Prepare dispersions of the test sample and the reference sample in an agreed medium. By mean of the film applicator or other device, apply to the substrate continuous films of both dispersions so that each is at least 25 mm wide (see Note). Allow to dry in a horizontal position in diffuse daylight for 24 h at normal room temperature. Alternatively, stove under the specified conditions. Cut test specimens of suitable size to fit the exposure frame, if used, so that the dividing line between the two dispersion films is central.

NOTE — If it is intended to carry out instrumental measurements, care shall be taken that the test specimen is of sufficient size.

12.2.2.2 Fasten a cover sheet across the middle one-third of the test specimen, clipping down the edges to prevent buckling, but in such a way that the cover may be removed for examination of the films and replaced in the same position.

12.2.2.3 The test specimen should be mounted behind window glass, parallel to it and at a distance of about 50 mm from it, protected from the weather, but with free circulation of air. It is desirable that the window glass should be

facing south in the northern hemisphere, and north in the southern hemisphere, and inclined to the horizontal at an angle equal to the latitude of the place of exposure.

12.2.2.4 Expose the test specimen to the source of light, and at suitable intervals of time examine it by raising the cover to determine if there is any change between the exposed and unexposed portions, replacing the cover in the same position. When the contrast between the exposed and unexposed portions of the reference sample is equal to that illustrated by Grade 4 of the geometric grey scale, record the degree of contrast of the film of the test sample and replace the cover.

12.2.2.5 Apply a further cover sheet so that two-thirds of the test specimen is covered and continue the exposure until the fully exposed and the central unexposed portions of the film of the reference sample show a contrast equal to Grade 3 of the geometric grey scale. Assess the amount of contrast or change of appearance of the film of the test sample in relation to that of the film of the test sample.

13 RELATIVE TINTING STRENGTH

13.0 Principle

13.0.1 A dispersion of the coloured pigment under test, prepared under known conditions on an automatic muller, is mixed in a known ratio with a dispersion of white pigment. The intensity of colour and hue of the resulting paste is compared with that of a similar paste made under the same conditions from the approved sample of coloured pigment and the same dispersion of white pigment.

13.0.2 The degree of development of tinting strength of a coloured pigment is dependent on the amount of work done in the preparation of the dispersion. Therefore, in determining the relative tinting strengths of two coloured pigments it is necessary for the comparison to be carried out at the level of maximum development of colour. In this method, which uses an automatic muller, the development of tinting strength is influenced by the force applied, the number of revolutions, the medium, the volume of the mix, and the rheology of the mix. By following the procedure described in **13.3.1**, the conditions are determined under which a practical maximum of tinting strength may be obtained on the automatic muller. When these conditions are known for a particular pigment, there is no need to carry out the procedure described in **13.3.1**.

13.0.3 Of the factors listed in **13.0.2**, the following can be specified without difficulty:

- The force applied, which shall be the maximum available;

- b) The medium to be used, which is linseed stand oil; and
- c) The volume of the mix of pigment and medium which shall be about 2 ml.

13.0.4 The remaining factors to be determined, which shall be chosen according to the level of tinting strength of the coloured pigment under test, are the following:

- a) The ratio of pigment to medium,
- b) The number of revolutions to be used, and
- c) The ratio of coloured pigment to white pigment.

13.0.5 The appropriate ratio of pigment to medium, by mass, depends not only on the oil absorption of the pigment but also on the viscosity of the mix during the mulling operation. As a first step, all pigments can initially be allocated to one of the following three groups:

- a) *Pigments of low medium requirement* — average mulling concentration 66.7 percent of pigment by mass;
- b) *Pigments of intermediate medium requirement* — average mulling concentration 40 percent of pigment by mass; and
- c) *Pigments of high medium requirement* — average mulling concentration 25 percent of pigment by mass.

13.0.6 The quantities to be used in the test for the three groups defined in 13.0.5 shall be the following:

- a) 3.0 g of pigment and 1.5 g of linseed stand oil;
- b) 1.0 g of pigment and 1.5 g of linseed stand oil; and
- c) 0.5 g of pigment and 1.5 g of linseed stand oil.

NOTES

- 1 If the mix chosen is found to be unsuitably stiff or fluid for use on the muller, one of the other ratios should be used as appropriate.
- 2 If the diameter of the muller plates is at the top end of the range specified in 13.1.1, it may be necessary to increase the amounts specified in order to reduce wear on the plates.

13.1 Apparatus

13.1.1 Automatic Muller

With ground glass plates, preferably water cooled, of diameter 180 to 250 mm to which a variable but known force of up to about 1 000 N may be applied. The driving glass plate shall have a speed of rotation of between 70 and 120 revolutions per minute and the apparatus shall have an arrangement for presetting the number of revolutions in multiples of 25.

13.1.2 Palette Knife or Spatula — Of steel or plastic material.

13.1.3 Glass Slides

Slides of glass or other transparent, colourless, non-absorbent substance.

13.1.4 Film of Plastics Material — Transparent and colourless.

13.1.5 Film Applicator

Suitable for applying two or three films side by side of wet paste, thickness 50 to 100 μm .

13.1.6 Balance — Accurate to ± 0.001 g.

13.2 Reagents

13.2.1 Linseed Stand Oil — (see Type 1 or 2 of IS 79 : 1975).

13.2.2 White Pigment Paste — With the following composition:

- 40 parts by mass of titanium dioxide, rutile (see IS 9788 : 1981)
- 60 parts by mass of linseed oil (see IS 75 : 1973).

13.2.2.1 Mix well so as to achieve preliminary wetting of the solids, then grind on a triple-roll mill, until the particle size is less than 5 μm when tested on a fineness of grind gauge. Store in airtight containers, preferably tubes with screw caps.

13.3 Procedure

13.3.1 Determination of Conditions for Preparing Dispersion of Coloured Pigments

13.3.1.1 Weigh an appropriate quantity of medium and transfer it to the lower plate of the muller; weigh the corresponding quantity of the approved sample of the coloured pigment and sprinkle it carefully on the medium. Mix the pigment with the medium by means of the spatula, but do not grind. Spread the mixture in an approximately circular patch, of diameter about 100 mm, in the centre of the lower plate and clean the spatula as much as possible by wiping it on the upper plate of the muller. Close the plates and carry out the grinding in stages of 50 revolutions; after each stage collect the paste from both plates with the spatula and spread it out in an approximately circular patch on the centre of the lower plate, wiping the spatula on the upper plate as before. After a total of 200 revolutions, remove a small portion (about one-quarter of the total volume) of the collected paste, set this aside and continue grinding the main bulk. Remove similar small portions after totals of 300 and 400 revolutions; set these also aside and then clean the muller plates and spatula.

13.3.1.2 Place on the lower muller plate 3 ± 0.01 g of white pigment paste and an amount of the coloured pigment paste which has been ground for 200 revolutions which contains 0.12 g of coloured pigment (see 13.3.1.4) and mix the two pastes with the spatula as homogeneously as possible, without grinding. Spread the mixture in an approximately circular patch in the centre of the lower plate of the muller, wipe the spatula on the upper plate, close the plates and grind for four stages of 25 revolutions each with the minimum applied force; after each stage collect the paste in the centre of the lower plate as described previously. Then remove the paste from the muller and set aside for later assessment. Repeat this operation with the same mass of each of the portions of coloured paste which have been ground for 300 and 400 revolutions, respectively.

13.3.1.3 Apply each of these reduction pastes in order, side by side, with touching edges on a sheet of glass or transparent film of plastics material so that the intensity of colour can be judged visually. Record the minimum number of revolutions necessary to obtain the sample of coloured pigment paste which produces the maximum intensity of colour and use the number of revolutions for the test itself.

13.3.1.4 The amount of coloured pigment paste which contains 0.12 g of coloured pigment will, when mixed with 3 g of white pigment paste, give a reduction ratio of 1 : 10. This ratio should be modified to for example, 1 : 5 or 1 : 20 (to suit weak pigments or strong pigments, respectively) in order to produce a colour on reduction of intensity which is suitable for assessing the intensity of colour and hue of the reduction pastes.

13.3.2 Preparation of Dispersion of Coloured Samples

Prepare a dispersion on the automatic muller of the approved sample of the coloured pigment as described in 13.3.1, but carrying out of the grinding in stages of 50 revolutions to the full number of revolutions previously decided without removing any of the paste, but gathering and spreading the paste after each stage. When the grinding has been completed, collect the paste and store. Clean the muller and spatula, and repeat the operation with the same quantities of the test sample of pigment and oil and using the same procedure on the same muller. Collect the coloured pigment paste from this sample and store. Clean the muller and spatula.

13.3.3 Mixing of Dispersions of Coloured Pigment and White Pigment

Place on the lower muller plate 3 ± 0.01 g of white pigment paste and an amount of the coloured pigment paste which has been ground

for 200 revolutions and which contains the mass of coloured pigment to give the chosen reduction ratio, and mix the two pastes with the spatula as homogeneously as possible, without grinding. Spread the mixture in an approximately circular patch in the centre of the lower plate, wipe the spatula on the upper plate, close the plates and grind for four stages of 25 revolutions each with the minimum applied force; after each stage collect the paste in the centre of the lower plate as described previously. Then remove the paste from the muller for later assessment and store in a suitable receptacle.

Repeat the operation using the coloured pigment dispersion paste prepared from the test sample of coloured pigment, thus producing a comparative reduction paste.

13.3.4 Comparison of Colour on Reduction and Determination of Relative Tinting Strength

13.3.4.1 Place a quantity of each of the two reduction pastes obtained as described in 13.3.3 side by side on a glass plate or plastics film, and with the film applicator draw them down to form two uniformly thick strips not less than 25 mm wide with touching edges not less than 40 mm long. Compare for intensity of colour and for hue the two strips immediately after application by examining them in diffused daylight through the glass or film of plastics material.

Where good daylight is not available, make the comparison in artificial light.

If the intensities of colour are equal and the hues are the same, the colours on reduction are the same, and the relative tinting strength of the sample under test is 100 percent.

However, if the intensities of colour are equal but the hues are not the same, and there is also a difference in colour on reduction; note the difference and its nature.

13.3.4.2 If the intensities of colour are not considered to be equal, and, therefore, the colours on reduction are not the same, repeat the operations of 13.3.3 and 13.3.4.1, but weighing and using a quantity of the coloured pigment dispersion of the test sample which is estimated to give an intensity of colour equal to that of the original mass taken of the coloured pigment dispersion of the approved sample.

For example, if it is considered that the sample under test is 15 percent stronger than the approved sample, in the repeat test, the mass of coloured test pigment dispersion used shall be 15 percent less than that taken for the first test, but the mass of coloured pigment dispersion of the approved sample shall be the same as that originally used. If there is also a difference in hue, note the difference and its nature.

13.4 Calculation

Relative tinting strength

$$= \frac{b \times 100}{a} \text{ percent of the approved sample}$$

where

a = parts of the test sample that produce the same intensity as approved sample, and

b = parts of the approved sample.

NOTE — It is important to include the percentage sign (%), and to note that the tinting strength is relative to that of the approved sample as 100 percent.

14 TEST FOR FREEDOM FROM ORGANIC COLOURING MATTERS

14.1 Procedure

Take 2 g of the material in a 100-ml conical flask, add 25 ml of 25 percent ethyl alcohol and stir well. Allow it to settle and carefully decant the supernatant liquid. Boil the residue with 25 ml of distilled water and decant the supernatant liquid. Boil the residue with 25 ml of dilute (1 : 4) ammonia and again decant the supernatant liquid. Boil another 2 g of the material with 25 ml of chloroform on electric waterbath. Allow it to settle and decant the supernatant liquid.

14.2 If any of the decanted supernatant liquid is coloured then organic colouring matter is present.

15 REDUCING POWER (OF WHITE PIGMENTS)

15.0 General

The reducing (lightening) power of white pigment is determined by treating the pigment with known quantity of the tinting media and comparing the same quantities of the approved sample with the same quantity of tinting media.

15.1 Apparatus — Same as given in 13.1.

15.2 Reagent

15.2.1 Tinting Paste

With the following composition;

— Castor oil, medicinal quality	: 500 g
— Precipitated calcium sulphate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$: 475 g
— Ultramarine blue (see IS 55 : 1970) or Phthalocyanine green	: 5 g
— Treated natural earth	: 20 g

The paste shall be prepared as follows:

'Mix the treated natural earth in a beaker with sufficient quantity of castor oil to give a uniform paste and then gradually stir in the remaining castor oil. Heat the mixture so obtained to a temperature of 50°C and after maintaining this temperature for about 15 minutes, stir in the ultramarine blue and calcium sulphate, adding them in small amounts. Disperse thoroughly the paste obtained, by passing through a roller mill or other suitable machine and stir to homogenize the paste thoroughly, heating, if necessary'.

Store the paste in airtight containers, preferably with screw caps.

15.3 Procedure

15.3.1 Incorporation of the White Pigment in to the Blue Paste by Means of Automatic Muller

Weigh, to the nearest mg, 5 g of the blue paste and place it in the middle of the clean lower plate of the muller. Weigh, to the nearest mg, the quantity (m_0) of the approved sample pigment indicated in Table 3 and incorporate it into the blue paste by gently working with the palette knife. When the white pigment has been wetted, spread the paste in a circle of approximately 50 mm diameter around the centre of the lower plate and clean the palette knife by drawing it across the top plate. Close the muller plates, apply a force of about 1 000 N and grind in four stages of 25 revolutions, picking up the paste with the same palette knife and transferring it to the centre of the plate after each stage.

When the grinding has been completed, remove the paste and store it on a palette.

15.3.2 Incorporation of the White Pigment into the Blue Paste by Means of a Hand Muller or Palette Knife

Weigh, to the nearest mg, 5 g of the blue paste and place it on the ground glass or marble plate. Weigh, to the nearest mg, the quantity (m_0) of the agreed sample pigment indicated in Table 3 and disperse it using the palette knife or a hand muller for 5 min with as little of the blue paste as possible, to obtain a smooth paste. Add the remainder of the tinting paste, a little at a time, to the rubbedout mixture and thoroughly mix with the palette knife, or hand muller, scraping up the paste frequently to ensure thorough mixing.

Remove the paste, as prepared, from the plate and store it on a palette.

15.3.3 Procedure for the Comparison

Treat the test sample in exactly the same manner as in 15.3.1 or 15.3.2 and determine the quantity

of pigment (m_1) which gives an intensity of colour equal to that of the paste of the agreed sample pigment.

Spread the two pastes made with the test sample and with the approved sample pigment in the same direction on the glass slide in opaque strips not less than 25 mm wide with touching edges not less than 40 mm long. Compare the pastes for intensity of colour by examining the two strips in diffuse daylight through the glass, and on the surface, immediately after application. Where good daylight is not available, make the comparison in artificial light.

Table 3 Approved Sample Pigment
(*Clauses 15.3.1 and 15.3.2*)

Pigment	Quantity to be Taken (m_0) g
Zinc oxide or lithopone 30 percent	0.500
High grade zinc sulphide	0.200
Titanium dioxide	0.100

15.4 Calculation

15.4.1 The reducing power of the test sample, taking the agreed sample as 100 :

$$= \frac{100 m_0}{m_1}$$

where

m_0 = mass, in g, of the agreed sample pigment, and

m_1 = mass, in g, of the test sample required to produce the same intensity of colour as the agreed sample pigment.

16 RELATIVE DENSITY

16.1 Apparatus

16.1.1 Pyknometer

Type 5 (see IS 5717 : 1990), of capacity 25 or 50 ml preferably with loose-fitting cap (see Fig. 2).

16.1.2 Vacuum Desiccator

Fitted with a two-hole stopper. A glass tube with a three-way stopcock passes through one hole of the stopper and connects the desiccator to the vacuum pump, and the stem of a dropping funnel passes through the other hole of the stopper.

16.1.3 Vacuum Pump

Vacuum pump or other apparatus which is capable of reducing the pressure to not greater than 2 kPa.

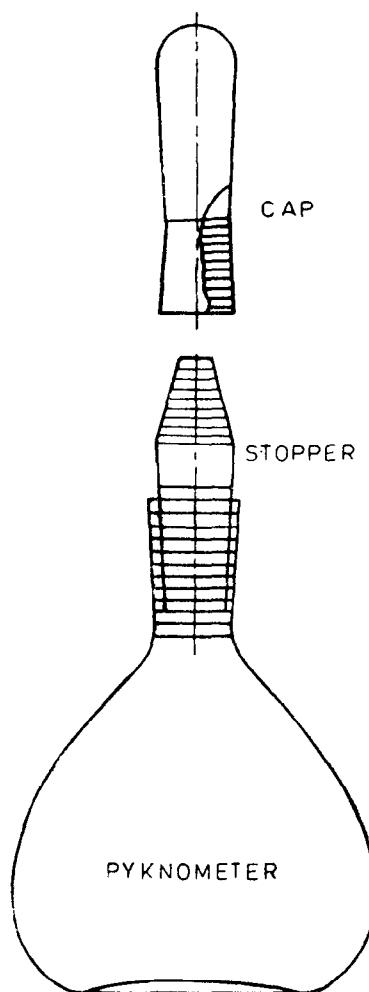


FIG. 2 PYKNOMETER GAS-LUSSAC TYPE

16.1.4 Water Bath

Thermostatically controlled, capable of being maintained to within $\pm 0.1^\circ\text{C}$ in the temperature range of 25 to 30°C (or at an agreed temperature t).

16.1.5 Balance — Accurate to 1 mg or better.

16.2 Reagent

A liquid should be selected in which the material to be tested is insoluble and which has good wetting properties and a low evaporation rate under a vacuum. High-boiling aromatic or aliphatic hydrocarbon solvents with final boiling point over 170°C are suitable. In addition to organic liquids, water with a wetting agent may also be selected.

16.3 Procedure

16.3.1 Clean and dry the pyknometer, stopper and cap. Fill the pyknometer with the liquid (see 16.2) and after allowing it to attain the

temperature of the bath, insert the stopper, wipe off the excess liquid, attach the cap and wipe dry the pyknometer. Transfer the pyknometer and the cap to the balance case, allow to stand for 15 min and weigh to the nearest mg.

16.3.2 Clean and dry the pyknometer, stopper and cap, and fill with distilled water. Carry out the procedure as in **16.3.1**.

16.3.3 Dry the pyknometer stopper and cap. Introduce by means of a dry funnel, a suitable quantity (1 to 10 g when the 25-ml pyknometer is used, or 2 to 20 g when the 50-ml pyknometer is used, depending on the density) of the dried sample so that the bottle is not more than half filled. Reweigh the stoppered pyknometer and cap.

16.3.4 Place the pyknometer containing the test portion in the vacuum desiccator and arrange the dropping funnel so that the stem of the funnel extends into the pyknometer. Close the stopcock of the dropping funnel and the three-way stopcock connecting the desiccator to the vacuum pump, start the pump and gradually open the three-way stopcock to the pump.

Fill the dropping funnel with liquid (see **16.2**) and, 15 min after the pressure in the desiccator has been reduced to not greater than 2 kPa, close the three-way stopcock and gradually open the stopcock of the funnel to admit sufficient liquid to cover the test portion completely to a depth of about 15 mm. Close the stopcock of the funnel and re-open the three-way stopcock to the pump, taking care to avoid losses by suction. Allow the pyknometer to remain in the desiccator under reduced pressure (not greater than 2 kPa) for about 4 h or until no air bubbles are visible in the liquid. Tap the desiccator occasionally to assist in removing entrapped air. Stop the pump and gradually open the three-way stopcock to admit air into the desiccator until the room pressure is restored.

16.3.5 Remove the pyknometer from the desiccator, fill it completely with the liquid and place it in the water bath maintained at $t \pm 0.1^\circ\text{C}$.

Allow the pyknometer to maintain for one hour in the bath in order to attain the temperature of the bath and then carefully insert the stopper so that the excess liquid fills the capillary. Wipe the liquid from the stopper. Remove the pyknometer from the bath, attach the cap and carefully wipe dry the pyknometer. Transfer the pyknometer and cap to the balance case, allow to stand for 15 min and weigh to the nearest mg.

16.4 Calculation

Calculate the density of the liquid (see **16.2**) at the temperature t by the equation:

$$\rho_1 = \frac{m_4 - m_1}{m_5 - m_1} \times \rho_0$$

and the relative density of the material tested by the equation:

$$\rho_m = \frac{\rho_1 (m_3 - m_1)}{(m_4 - m_1) - (m_3 - m_2)}$$

where

m_1 = mass, in g, of the pyknometer, stopper and cap;

m_2 = mass, in g, of the pyknometer, stopper, cap and sample;

m_3 = mass, in g, of the pyknometer, stopper, cap, sample and liquid;

m_4 = mass, in g, of the pyknometer, stopper, cap and liquid;

m_5 = mass, in g, of the pyknometer, stopper, cap and distilled water;

ρ_0 = density, in g per ml, of the water at the temperature t (see Table 4 for values at different temperatures); and

ρ_1 = density, in g per ml, of the liquid at temperature t .

Table 4 Density of Water at 15-30°C

Temperature of Water, t°C	Density of Water ρ_0 , g/ml
15	0.999 1
20	0.998 2
25	0.997 0
30	0.995 6

17 BULK DENSITY

17.1 Apparatus

17.1.1 Assemble the apparatus as shown in Fig. 3. The measuring cylinder *A* shall be of 250-ml capacity (see IS 878 : 1975). The base of the measuring cylinder shall be ground flat. The distance between the flat-ground part of the base of the measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height, shall be 25 ± 2 mm.

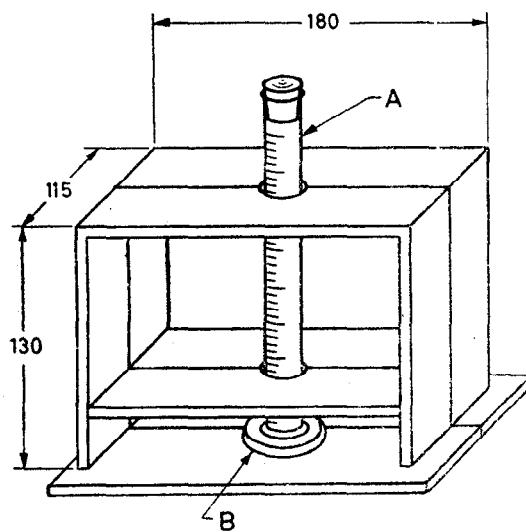
17.1.2 The rubber base pad *B* shall have a shore hardness of 42 to 50.

17.1.3 Pans of the balance shall be at least 10 cm in diameter and the balance shall have an accuracy of 0.1 g or better.

17.2 Procedure

Take sufficient material so as to make up the volume up to 100 ml mark. Dry, sieve the material through 250-micron IS Sieve by lightly

brushing with a 25 mm wide and 12.5 mm thick good quality paint brush on to a tared glazed paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder, which should be held at 45° to the vertical, without knocking or squeezing. Assemble the apparatus as shown in Fig. 3. With the thumb and fingers of one hand, gently grasp the upper part of the cylinder, and within one second, lift it as far as 25 mm (see Fig. 3). Do not jerk the cylinder by knocking it against the upper stop. At the start of the next second, release the cylinder smoothly. Continue lifting and dropping at the rate of one drop per second until 50 complete drops have been given. Once every two seconds, a gentle twist of about 10° should be given to the cylinder. As soon as 50 drops have been completed, raise the cylinder to eye level and read the volume of the sieved material.



All dimensions in millimetres.

FIG. 3 APPARATUS FOR THE DETERMINATION OF BULK DENSITY

17.3 Calculation

$$\text{Bulk density, g/l} = \frac{M}{V}$$

where

M = mass, in g, of the sieved material taken for test; and

V = final volume, in litres, of the sieved material.

18 PARTICLE SHAPE

18.0 General

Prepare a thin film of the material on a glass plate and view through a microscope at 400 magnification.

18.1 Apparatus

18.1.1 Microscope

Suitable for viewing at minimum of 400 magnification.

18.2 Procedure

Mix a small quantity, about 0.1 g, of the material with petroleum hydrocarbon solvent and apply a thin film of the paste on a clean glass slide. The film should be free from any aggregate particles as far as possible. Observe the shape of the particles under the microscope using 400 magnification. Compare the shape of the particles with a similarly prepared slide of the approved sample. The shape of the particles shall not differ from that of the approved sample.

19 MATTER SOLUBLE IN WATER

19.0 General

Two methods (hot extraction and cold extraction) are prescribed. The choice of the method for testing individual inorganic pigment and extender shall be as specified in the material specification.

19.1 Apparatus

19.1.1 *One-Mark Volumetric Flask* — Of capacity 250 ml.

19.1.2 *Colloid Filter*

19.1.3 *Evaporating Dish* — Flat bottomed, of glass, platinum, glazed porcelain or silica.

19.1.4 *Oven* — Capable of being maintained at $150 \pm 2^\circ\text{C}$.

19.1.5 *Balance* — Accurate to 1 mg or better.

19.1.6 *Desiccator*

19.2 Reagent

19.2.1 *Distilled Water*

Fresh, double distilled or de-ionized water, of pH 6 to 7.

19.3 Procedure

19.3.1 *Hot Extraction Method*

Weigh 2 to 20 g of the sample, to the nearest 0.01 g, into a beaker. Wet the sample in the beaker with a few ml of the water. Add 200 ml of the water and stir. Unless a different period is specified for the material under test, boil the water for 5 min. A coagulating agent may be used if the semi-colloidal nature of the material makes this desirable, provided that the agent selected is not such as to affect the subsequent determination of the acidity or alkalinity of the aqueous extract, and provided that only the minimum quantity is used.

Cool rapidly to room temperature, transfer to the volumetric flask and dilute to the mark with water. Mix thoroughly by shaking and inversion, and filter through the colloid filter, returning

the filtrate to the filter until it runs clear. Evaporate 100 ml of the perfectly clear filtrate to dryness in the previously weighed evaporating dish on a water bath.

Dry the residue in the evaporating dish in the oven at $105 \pm 2^\circ\text{C}$, cool in the desiccator and weigh to the nearest mg. Repeat the heating and cooling until the results of the two consecutive weighings, at an interval including at least 30 min of heating, do not differ by more than 10 percent of the final figure obtained for the water soluble matter.

19.3.2 Cold Extraction Method

Weigh 2 to 20 g of sample, to the nearest 0.01 g, into a beaker. Wet the sample in the beaker with a few ml of water.

NOTE — If the material does not disperse easily in water, a wetting agent should be used. In the case of materials not soluble in ethanol, 5 ml of ethanol may be used; in the case of pigments soluble in ethanol, a non-ionic wetting agent such as 10 ml of a 0.01 percent solution of an ethylene oxide condensate should be used. If the wetting agent is non-volatile under the conditions of test, an appropriate correction derived from a blank test should be made.

Add 200 ml of the water (cooled to room temperature) and stir continuously for one hour at room temperature. Transfer to the volumetric flask and dilute to the mark with water. Mix thoroughly by shaking and inversion, and filter through the colloid filter, returning the filtrate to the filter until it runs clear. Evaporate 100 ml of the perfectly clear filtrate to dryness, in the previously weighed evaporating dish, on a water bath.

Dry the residue in the evaporating dish in the oven at $105 \pm 2^\circ\text{C}$, cool in the desiccator and weigh to the nearest mg. Repeat the heating and cooling until the results of the two consecutive weighings, at an interval including at least 30 min of heating, do not differ by more than 10 percent of the final value obtained for the water soluble matter.

19.4 Calculation

Water soluble matter, percent by mass = $\frac{250 m_1}{m_0}$
where

m_0 = mass, in g, of the sample; and

m_1 = mass, in g, of residue.

20 ACIDITY AND ALKALINITY

20.1 Apparatus

20.1.1 *Burette* — 50 ml capacity (see IS 1997 : 1982).

20.1.2 *pH Measuring Device* — Capable of measurement to 0.1 unit, calibrated against buffer solutions of known pH value at the temperature of the test.

20.2 Reagents

20.2.1 *Hydrochloric Acid* — 0.05 N.

20.2.2 *Sodium or Potassium Hydroxide* — 0.05 N.

20.2.3 *Methyl Red Indicator* — 1 g/l solution in 60 percent (v/v) ethanol.

20.3 Procedure

20.3.1 Dissolve the sample into water and make the solution as in 19.3.1.

NOTE — If the filtrate is coloured, the use of indicator is not suitable. In that case, the potentiometric method should be used.

20.3.2 With Indicator Solution (Method A)

Add 5 drops of the methyl red indicator to 100 ml of the test solution (20.3.1). If the solution is orange, consider it as being neutral. If the solution is yellow (alkaline), titrate it with the hydrochloric acid solution to an orange end point. If the solution is red (acidic), titrate it with the sodium hydroxide solution to an orange end point.

NOTE — By agreement between the parties, another colour indicator may be used.

20.3.3 Potentiometric Determination (Method B)

Take 100 ml of the test solution (20.3.1), insert the electrodes of the pH measuring device and read the pH value. If the pH value is between 4 and 8, consider the solution as being neutral. If the pH value is more than 8 (alkaline), titrate the solution with the hydrochloric acid solution to a pH value just less than 8. If the pH value is less than 4 (acid), titrate the solution with the sodium or potassium hydroxide solution to a pH value just more than 4.

20.4 Calculation

Acidity (alkalinity), ml of 0.1 N acid (alkali) for 100 g of sample

$$= \frac{V \times 2.5 \times 100}{2 \times m} = 125 \frac{V}{m}$$

where

m = mass, in g, of the sample taken to prepare the test solution (20.3.1); and

V = volume, in ml, of the sodium hydroxide solution or hydrochloric acid solution.

If the extract is neutral, report the result as 'neutral'.

21 pH VALUE

21.1 Apparatus

21.1.1 Glass Container

Capacity 50 ml, made of chemically resistant glass, fitted with a ground glass or rubber stopper.

NOTE — The rubber stopper shall not have been used for any other purpose.

21.1.2 pH Measuring Device

Capable of measurement to 0.1 unit, calibrated against buffer solutions of known pH value at the temperature of the test.

21.1.3 Balance — With an appropriate accuracy.

21.2 Procedure

Prepare a 10 percent (m/m) suspension of the material under test, using distilled water, in the glass container. Stopper the container and shake it vigorously for one minute. Allow to stand for 5 min, remove the stopper and determine, to the nearest 0.1 unit, the pH value of the suspension.

NOTE — If the material does not disperse easily in water, a wetting agent should be used; in the case of materials not soluble in ethanol, up to 5 ml of ethanol may be used but care should be taken to ensure that the minimum quantity is used and that it is neutral and free from pyridine. In the case of pigments soluble in ethanol, a neutral non-ionic wetting agent, such as 10 ml of a 0.01 percent (m/m) solution of an ethylene oxide condensate, should be used. The neutrality of the wetting agents should be checked by making a blank determination. If a wetting agent is used, the volume of the water should be reduced so that a 10 percent (m/m) suspension is obtained.

Record the pH value to the nearest 0.1 unit and the temperature of the suspension to the nearest 1°C. If the duplicate determinations of pH differ by more than 0.3 unit, the procedure shall be repeated.

22 WATER-SOLUBLE SULPHATES, CHLORIDES AND NITRATES

22.1 Apparatus

22.1.1 *Sintered Glass Crucible* — Porosity grade (pore size index 10-16 μm) (see IS 12305 : 1988).

22.1.2 *Nessler Cylinders* — Capacity 50 ml.

22.1.3 *Distillation Apparatus*

22.2 Reagents

22.2.1 *Concentrated Hydrochloric Acid* — Sp. gravity 1.18.

22.2.2 *Silver Nitrate* — 0.01 N standard volumetric solution (see IS 2316 : 1968).

22.2.3 *Ammonium Chloride Solution* — 17.2 mg/l.

22.2.4 *Sodium Hydroxide Solution* — 200 g/l.

22.2.5 *Barium Chloride Solution* — 50 g/l.

22.2.6 *Potassium Chromate Solution* — 50 g/l.

22.2.7 *Devarda's Alloy* — Powdered.

22.2.8 *Nessler's Reagents*

Prepared by either method (a) or method (b) as follows:

- Dissolve 5 g of potassium iodide in 3.5 ml of water. Add cold saturated mercuric chloride (HgCl_2) solution, while stirring, until a faint red precipitate is formed. Continue stirring, add 40 ml of potassium hydroxide solution (500 g/l), dilute to 100 ml and mix well. Allow to settle, decant the clear supernatant liquid and store in the dark.
- Dissolve 3.5 g of potassium iodide and 1.25 g of mercuric chloride in 80 ml of water. Add cold saturated mercuric chloride solution, while shaking, until a slight red precipitate remains, then add 12 g of sodium hydroxide, shake until dissolved, and finally add a little more of the saturated mercuric chloride solution and dilute to 100 ml with water. Shake occasionally during several days, allow to stand, and use the clear supernatant liquid for the test.

22.3 Determination of Sulphates

22.3.1 Procedure

Take 50 ml of the clear aqueous extract obtained in 19.3.1 or 19.3.2, acidify with 3 ml of the hydrochloric acid and boil the solution vigorously, taking care to avoid loss of solution by splashing. Add the barium chloride solution, drop by drop, to the hot solution until in slight excess, and allow the solution to stand overnight. Decant the supernatant liquid through the tared sintered glass crucible, transfer the precipitate to the crucible of porosity grade 16 and wash it free from chloride. Ignite it gently, then at red heat, cool it in a desiccator and weigh to the nearest mg.

22.3.2 Calculation

$$\text{Water-soluble sulphate (as } \text{SO}_4 \text{), } = \frac{206 m_1}{m_0} \text{ percent by mass}$$

where

m_0 = mass, in g, of pigment used in the determination of matter soluble in water; and

m_1 = mass, in g, of barium sulphate precipitate.

22.4 Determination of Chlorides

22.4.1 Procedure

Take 50 ml of the clear aqueous extract obtained in 19.3.1 or 19.3.2 and add 1 ml of the potassium chromate solution. Titrate with the silver nitrate solution, slowly and with vigorous shaking, until a faint reddish brown colour persists.

Carry out a blank determination by adding 1 ml of potassium chromate solution to 50 ml of water and titrate with the silver nitrate solution until the colour matches that of the previous titration making due allowance for any opalescence or turbidity.

22.4.2 Calculation

Water soluble chloride (as Cl), percent by mass

$$= 0.1775 \frac{(V_1 - V_0)}{m}$$

where

V_0 = volume, in ml, of 0.01 N silver nitrate solution required for the blank determination;

V_1 = volume, in ml, of 0.01 N silver nitrate solution required by the sample; and

m = mass, in g, of pigment used in the determination of matter soluble in water.

22.5 Determination of Nitrates

22.5.1 Procedure

Place 50 ml of the clear aqueous extract obtained in 19.3.1 or 19.3.2 in the distillation flask and dilute to 150 ml. Add 3 g of Devarda's alloy and 30 ml of the sodium hydroxide solution and close the apparatus at once. Place 2 ml of the hydrochloric acid and 30 ml of water in the receiver. Warm the flask gently until the reaction starts and then allow the reaction to proceed gently for about half an hour. Then distil about 70 ml of liquid, the receiver being kept cool with running water. Make up the distillate to 250 ml with water and transfer 5 ml to a Nessler cylinder. Dilute to 50 ml, add 1 ml of Nessler's reagent and match the colour against that of a similar standard solution prepared by adding ammonium chloride solution from a burette. Carry out a blank determination using 50 ml of distilled water.

22.5.2 Calculation

Water soluble nitrate (as NO_3), percent by mass

$$= 0.5 \frac{(V_1 - V_0)}{m}$$

where

V_0 = volume, in ml, of ammonium chloride solution required by the blank determination;

V_1 = volume, in ml, of ammonium chloride solution required by the test portion; and

m = mass, in g, of pigment used in the determination of matter soluble in water.

23 ETHER EXTRACT

23.1 Procedure

Weigh accurately about 10 g of the dried material in an extraction thimble with either a filter paper or a plug of glass wool. Extract with solvent ether (see IS 336 : 1973), the material contained in the thimble in a Soxhlet apparatus for at least 2 h, during which time the solvent should siphon at least 8 times per hour. Remove the volatile solvent from the extract in the extraction flask until constant mass is obtained. Express the ether solubles as the percent of the material taken for test.

24 ASH CONTENT

24.1 The quantity of pigment to be taken for this shall be such as to give a weighable residue.

24.2 Procedure

Incinerate the accurately weighed quantity of the dried material to constant mass at dull red heat with free access to air. From the difference in mass, calculate the result as percentage of the mass of the material taken for test.

25 LEAD RESTRICTION

25.1 Procedure

Shake a weighed quantity of the dried material with 1 000 times its mass of dilute hydrochloric acid (0.25 percent) continuously for one hour at room temperature. Allow the mixture to stand for one hour and then filter. Precipitate the lead salt contained in the clear filtrate as lead sulphate using sulphuric acid. Transfer the precipitate and filter paper to a tared silica crucible, dry in an oven and carefully ignite adding 1 to 2 drops of concentrated sulphuric acid to the ash, cool and weigh as lead sulphate. Calculate and report as lead monoxide (PbO).

25.2 Alternatively, determine the quantity of lead by electrolysis after dissolving the precipitated lead sulphide in nitric acid, and calculate as lead monoxide (PbO). Express the result as percent of the mass of the material taken for test.

26 FREEDOM FROM LEAD

26.1 Reagents

26.1.1 *Dilute Nitric Acid* — Lead-free, approximately 4 N.

26.1.2 *Ammonium Acetate Solution* — Lead-free, 10 percent (*m/v*).

26.1.3 *Concentrated Sulphuric Acid* — Lead-free, *rd* 1.84.

26.1.4 Dilute Sulphuric Acid

Two percent, obtained by dissolving 11 ml of concentrated sulphuric acid in one litre of water.

26.1.5 Ammonium Acetate — Solid, lead-free.**26.1.6 Hydrogen Sulphide — Gas, from Kipps apparatus.****26.1.7 Hydrogen Sulphide Water — Saturated solution.****26.2 Procedure**

26.2.1 Treat about 20 g of the dried material, accurately weighed, with dilute nitric acid. The quantity of acid is immaterial provided it is sufficient to extract the soluble matter; but avoid too great an excess since it has to be evaporated off. Allow the basin to stand on a boiling water bath for at least three hours. In case a large quantity of insoluble residue is left, heat the basin on the water bath overnight. Decant off the supernatant liquid through a filter paper and extract the insoluble residue again on a boiling water bath for one hour with dilute nitric acid. Filter through the same filter paper and wash the residue thoroughly on the filter paper with hot water. Treat the residue on the filter paper with 10 ml of ammonium acetate solution, filter and wash again.

26.2.2 Mix the filtrates and washings in a 500-ml evaporating basin, add 2 ml of concentrated sulphuric acid, and evaporate the contents of the basin on a sand-bath till fumes appear. Add 100 ml of water to the basin and allow to stand on the boiling water bath for 15 minutes. Then dilute the contents to about 150 ml and allow to stand overnight at room temperature. Filter the insoluble matter on a Whatman No. 42 filter paper or its equivalent (9 cm) and wash thoroughly with dilute sulphuric acid.

26.2.3 Transfer the filter paper and residue to a small beaker, cover with 20 ml of water, and add 1 to 2 g of solid ammonium acetate. Heat the beaker on the water bath for not less than half an hour, stirring the contents occasionally. Decant the liquid through a Whatman No. 42 filter paper or its equivalent (9 cm). Repeat the extraction with water and ammonium acetate. Transfer all the insoluble matter including the filter pulp to the filter paper and wash thoroughly with warm water, collecting the filtrate and washings in a 150-ml beaker. Pass hydrogen sulphide through the liquid for 10 to 15 min and filter the precipitated lead sulphide at once thorough a No. 40 Whatman filter paper or its equivalent (9 cm). Wash thoroughly but quickly

with hydrogen sulphide water, keeping the residue in the filter paper, if any, covered with liquid till the washing is completed.

26.2.4 Transfer the precipitate and filter paper to a tared silica crucible. Dry in an oven and carefully ignite adding 1 to 2 drops of concentrated sulphuric acid to ash, cool and weigh as lead sulphate. From the difference in mass of the crucible, calculate the mass of lead and express as percent of mass of the material taken for test.

26.3 Alternatively, determine the quantity of lead by electrolysis after dissolving the precipitated lead sulphide in nitric acid.

26.4 Calculation

Calculate as lead (Pb) and express the result as the percent of the mass of the material taken for test.

27 MARKING AND DELIVERY

27.1 In all cases, the indication of the source of manufacture, batch number and the month and year of manufacture shall be marked on each container.

27.2 Marking and delivery shall be in accordance with the instructions given by the purchaser, subject to the provisions of **27.2.1** and **27.2.2**.

27.2.1 Lead Pigments

If the material is a lead pigment, each container in which the pigment is supplied shall be legibly and indelibly marked 'LEAD PIGMENT' in letters not less than 25 mm high.

27.2.2 Lead-Free Pigments

In no part of the package shall the amount of lead or lead compounds or both of them, calculated as metallic lead, exceed 0.5 percent. This restriction applies to the material used in the construction of packages, any metallic coating thereof, and any solder used for manufacture, repair or sealing. In addition to the marking, as required by the purchaser, all containers containing the pigment shall be clearly marked 'LEAD-FREE' in black or white letters, whichever is more legible, the letters being not less than 25 mm high. Paint or other material used for marking and painting the packages, when required, shall not contain lead exceeding 0.03 percent.

ANNEX A

(Clause 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	Title	IS No.	Title
55 : 1970	Ultramarine blue for paints (<i>first revision</i>)	878 : 1975	Graduated measuring cylinders (<i>first revision</i>)
75 : 1973	Linseed oil, raw and refined (<i>second revision</i>)	1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
77 : 1976	Linseed oil, boiled for paints (<i>second revision</i>)	1303 : 1983	Glossary of terms relating to paints (<i>second revision</i>)
79 : 1975	Linseed stand oil for paints (<i>second revision</i>)	1745 : 1978	Petroleum hydrocarbon solvents (<i>second revision</i>)
196 : 1966	Atmospheric conditions for testing (<i>revised</i>)	1997 : 1982	Burettes (<i>second revision</i>)
385 : 1979	Mixed liquid driers for paints (<i>second revision</i>)	2316 : 1968	Methods of preparation of standard solutions for colorimetric and volumetric analysis (<i>first revision</i>)
460 (Part 2) : 1985	Test sieve : Part 2 Perforated plate test sieves (<i>third revision</i>)	5717 : 1970	Pyknometers
768 : 1982	Method for evaluating change in colour (<i>first revision</i>)	9788 : 1981	Titanium dioxide, rutile for paints
		12305 : 1988	Laboratory sintered (fritted) filters, porosity grading, classification and designation

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Amendments Issued Since Publication

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